

## Low friction $\text{MoS}_2(\text{Ti}, \text{W})$ coatings with a Ti interlayer, magnetron sputtered on silumin

J. Gawroński <sup>a,\*</sup>, P. Nolbrzak <sup>b</sup>, G. Gumienny <sup>a</sup>

<sup>a</sup> Department of Material Engineering and Production Systems, Faculty of Mechanical Engineering, Lodz University of Technology, ul. Stefanowskiego 1/15, 90-924 Łódź, Poland

<sup>b</sup> Institute of Materials Science and Engineering, Faculty of Mechanical Engineering, Lodz University of Technology, ul. Stefanowskiego 1/15, 90-924 Łódź, Poland

\* Corresponding e-mail address: jakub.gawronski@p.lodz.pl

### ABSTRACT

**Purpose:** The paper presents results of the selected mechanical and tribological properties of the low friction  $\text{MoS}_2(\text{Ti}, \text{W})$  coatings with a Ti interlayer deposited by magnetron sputtering method on silumin.

**Design/methodology/approach:**  $\text{MoS}_2(\text{Ti}, \text{W})$  coating was investigated by basic tribological tests and it was characterized by linear distribution analysis of selected chemical elements (EDS), nanohardness and Young's modulus.

**Findings:** It was established that the total thickness of the produced  $\text{MoS}_2(\text{Ti}, \text{W})$  coatings with the Ti layer equaled about 2.5  $\mu\text{m}$ . The  $\text{MoS}_2(\text{Ti}, \text{W})$  layers produced on a substrate significantly increase its functional properties, by significantly lowering the friction coefficient and wear, thus improving its mechanical properties (hardness and Young's modulus).

**Practical implications:** Silumins used as a substrates should be previously refined before coating process to reduce the porosity of the material and to obtain maximum adherence of  $\text{MoS}_2(\text{Ti}, \text{W})$  layer.

**Originality/value:** The increasingly accurate knowledge on the subject of the relations between the composition, processing, microstructure characteristics and properties has led to the improvement of aluminium properties. However, there is the need to improve of the tribological properties of these alloys what authors obtained.

**Keywords:** Friction; Silumin; MoS coating

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### PROPERTIES

## 1. Introduction

The increasingly frequent use of aluminium alloys in the broadly understood transport and armaments industry from their special properties, such as low density, high corrosion resistance, elevated plasticity at lowered temperatures and, most of all, high strength/mass ratio, which enables a significant reduction of the vehicle's mass and thus a reduction of fuel consumption and CO<sub>2</sub> emission with simultaneous increase of their scope. This perfectly reflects the strategy of the Kyoto document, providing for a reduction of CO<sub>2</sub> emission into the atmosphere. Unfortunately, aluminium-based alloys characterize in low tribological properties and a modification of their surface is necessary. The expected results can be obtained with the use of the PVD and CVD surface modification methods [1-3], which allow for obtaining basically any coating and make it possible to control their properties during the deposition process. They include: mono- or multi-layer coatings [4-6], gradient [5,7-9] and nano-composite [5,10-16] coatings, coatings for friction couples working at temperatures above 300°C [17-19], as well as carbon-based [13,20-24] and molybdenum disulfide coatings [14,25].

## 2. Materials and work methodology

The authors decided to apply magnetron sputtering to produce a low friction MoS<sub>2</sub> (Ti,W) coating with a Ti interlayer on sample of silumin which was refined beforehand. The task of the Ti interlayer was to increase the adhesion of the coating to the substrate. The basic mechanical properties and the wear resistance of the produced coatings were investigated.

The chemical composition of the substrate was determined by X-ray spectroscopy, and it corresponds to the French standard EN AC-47100. Its results are included in Table 1.

Table 1.  
Chemical composition of samples made of ENAC-47100 silumin

Chemical composition, %										
Si	Fe	Cu	Mn	Mg	Cr	Ni	Zn	Pb	rest	
10.5	1.3	1.0	0.55	0.35	0.10	0.30	0.55	0.20	0.25	

The coated samples' dimensions were 10x10x0.5 mm. Before the process of applying the coatings, the samples were ground with abrasive papers of a gradually increasing

gradation, up to 2000. Next, they were polished by means of a polishing wheel with diamond powder, fraction 1 µm. The prepared samples were then washed in detergent and acetone with the use of an ultrasonic washer, and then, after they became dry, they were mounted in the chamber of the device prepared for the coating production.

Metallographic tests were performed on transverse microsections with the use of the scanning microscope JEOL JSM-6610LV with integrated MiniCL-GATAN Cathodoluminescence Imaging and Oxford Instruments systems. A linear X-ray microanalysis of selected elements in the nanocomposite MoS<sub>2</sub>(Ti,W)+Ti coating was conducted by means of the EDS X-MAX 80 attachment and the back-scattered electron acquisition system EBSD NordlysMax installed in JEOL JSM-6610LV by EDS. The tests were performed with the use of the EDS AZtecEnergy and EBSD AztecHKL software. The parameters of the beam were selected in such a way so that the depth of its penetration would not be higher than the thickness of the produced coating.

Tests of the nanohardness and Young's modulus of the produced coating were conducted with the use of the nanoindenter F-MY MTS Instruments Nano G-200.

Tests of the adhesion of the coating to the substrate were also performed with the use of the nanoindenter F-MY MTS Instruments Nano G-200 by way of a scratch test. This method consists in a horizontal shift of a penetrator tipped with a diamond cone of the apex angle 87.7° and the cone radius 0.91 µm, pressed by the axial force towards the sample surface with the produced coating and changing in the range from 0.1 mN to 40 mN at the measuring length from 0 to 500 nm (1 mN/80 nm). The primary load for the preliminary section equaled 0.1 mN. The speed of the penetrator shift was 10 µm/sec. The adhesion assessment is performed on the basis of the observations of the changes in the coefficient of friction.

Friction and wear measurements were made in the 'pin-on-disc' geometry with the use of the CSM device, Switzerland. The counter-sample made of bearing steel 100Cr6 was 6.35 mm in diameter. The coefficient of dry friction was measured with the load of 1 N and with the friction radius of 8.5 mm. The linear friction velocity equaled 0.1 m/s. The test path of friction was 1000 m. The tribological tests were performed with the relative humidity of 40±1% at room temperature.

After the friction tests, transverse microsections of the wear tracks were made with the use of the profilometer Hommel Tester T1000 in order to determine the wear coefficient  $K_w = V/F \cdot l$ , where  $K_w$  – coefficient of friction ( $m^3 \cdot N^{-1} \cdot m^{-1}$ );  $V$  – volume worn during the friction tests in

the wear track ( $\text{m}^3$ );  $F$  – friction couple load (N);  $l$  – path of friction (m).

The deposition of the  $\text{MoS}_2(\text{Ti}, \text{W})$  coatings with the Ti interlayer was performed in the reaction chamber of the vacuum coater B-90 (Fig. 1), with the use of four magnetron sources: two equipped with targets from the  $\text{MoS}_2$  sinter with the addition of Ti (12% at.), one with a target from metallic W and one with a target from metallic Ti.

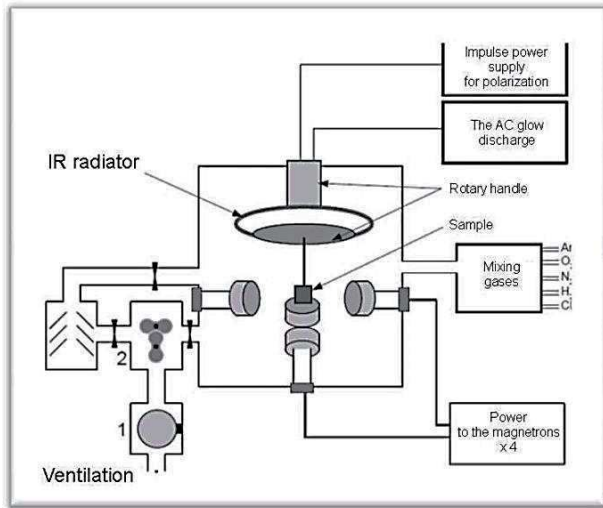


Fig. 1. The scheme of the  $\text{MoS}_2(\text{Ti}, \text{W})$  coating device with the Ti interlayer

Before the coating deposition process, the residual pressure in the working chamber was pumped away to the value  $<1.5 \cdot 10^{-3}$  Pa. This was followed by cleaning of the coated substrates with the use of glow discharge plasma. Etching of the surfaces was conducted under the pressure of 3 Pa for the time of 720 s. After the cleaning procedure was finished, the argon flow was closed and the chamber was pumped away to the pressure of  $<1.5 \cdot 10^{-3}$  Pa.

During the deposition of the  $\text{MoS}_2(\text{Ti}, \text{W})$  coatings, the variant with the Ti interlayer was applied, with the purpose to compare the obtained adhesion values of a similar base coating with a Cr interlayer [16].

Before the deposition process began, argon was introduced into the reaction chamber, and the pressure was set at the level of 0.45 Pa. The substrates were polarized with the negative voltage of 50 V. The time of deposition of the metallic interlayer equaled 600 s. The process consisted of two stages. In the first stage, a metallic Ti layer was applied (time 300s), and in the second stage, a magnetron with a W disk and magnetrons with  $\text{MoS}_2+\text{Ti}$  targets were activated. The power on the magnetrons with

the  $\text{MoS}_2+\text{Ti}$  disks was being increased for the following 300 s, from 0 to 1.6 kW (on each of them). After 600 s from the beginning of the sputtering of the metallic Ti disk, the magnetron was deactivated and the process of main layer deposition began, with the participation of two  $\text{MoS}_2+\text{Ti}$  12 at% disk magnetrons and one metallic T disk magnetron, which lasted 4500 s. During the whole process of interlayer and main layer deposition, the coated substrates were placed in a holder rotating in the symmetry axis of the chamber, at the frequency of 0.2 turns/s (12 turns/min), and the temperature on their surface did not exceed  $150^\circ\text{C}$ .

An important parameter was the substrate temperature, which, during the deposition process, could not exceed  $150^\circ\text{C}$ .

### 3. Results

With the use of a scanning electron microscope, it was established that the total thickness of the produced  $\text{MoS}_2(\text{Ti}, \text{W})$  coatings with the Ti layer equaled about  $2.5 \mu\text{m}$  (Fig. 2a). The thickness of the titanium layer was determined to be about 200nm, which was confirmed by the X-ray microanalysis tests (Fig. 2b), performed along the marked line crossing the section of the coating (Fig. 2a).

The produced  $\text{MoS}_2(\text{Ti}, \text{W})$  coating characterizes in the hardness of 6.5-7 GPa (Fig. 3) and the Young's modulus of about 85 GPa (Fig. 4).

Figures 5 and 6 show the changes in the averaged coefficient of friction during the adhesion measurement of the  $\text{MoS}_2(\text{Ti}, \text{W})$  coating only (Fig. 5) and the  $\text{MoS}_2(\text{Ti}, \text{W})$  coating with the Ti layer (Fig. 6), for the load of 1 N. A significant change in the adhesion of the  $\text{MoS}_2(\text{Ti}, \text{W})$  coating with the Ti layer (Fig. 6) took place with the load of  $\approx 10$ -12 mN, which, compared to the adhesion of the coating without the interlayer, i.e.  $\approx 7$ -8 mN, points to its significant increase, fulfilling the assumptions of the research.

The friction tests with the recording of the dynamic coefficient of friction showed that the coefficient of friction for the silumin without the coating oscillates around 0.5, whereas the one with the  $\text{MoS}_2(\text{Ti}, \text{W})$  coating is much lower and equals about 0.08 (Fig. 7).

The Kw coefficients of wear seem to be very interesting (Table 2), as they show that the silumin sample without the produced coating wears much faster than the silumin with the  $\text{MoS}_2(\text{Ti}, \text{W})$  coating. These coefficients equal:  $2.4 \cdot 10^{-13}$  for the silumin without the coating and  $8.5 \cdot 10^{-15}$  for the silumin with the coating.

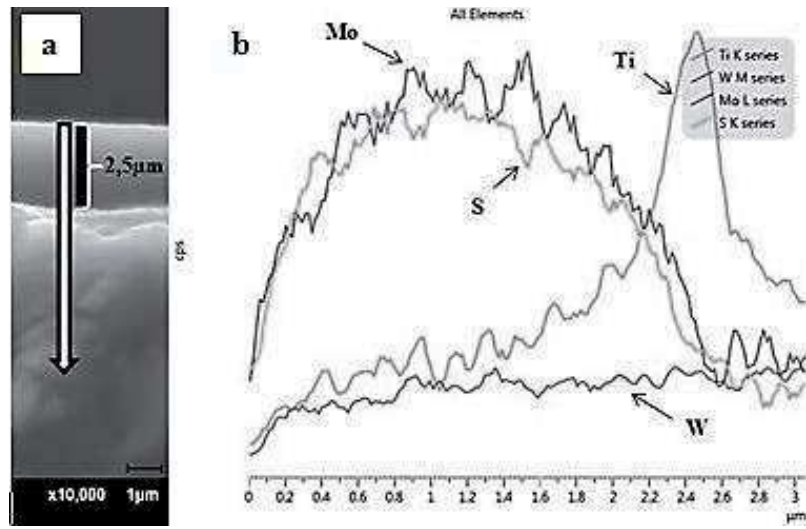


Fig. 2. MoS<sub>2</sub>(Ti,W) coating with Ti interlayer deposited on silumin (a) and microanalysis along the line across the coating (b) (content of Ti, W, Mo, S)

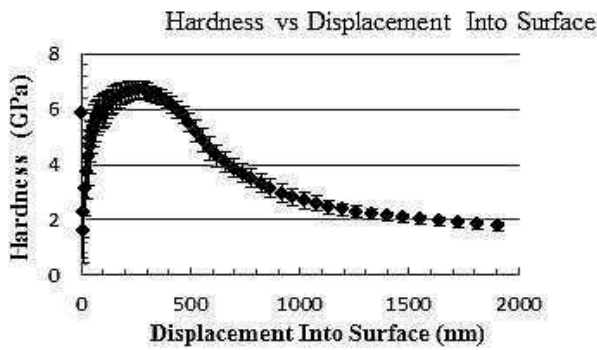


Fig. 3. Nanohardness diagram of MoS<sub>2</sub>(Ti,W) with Ti interlayer

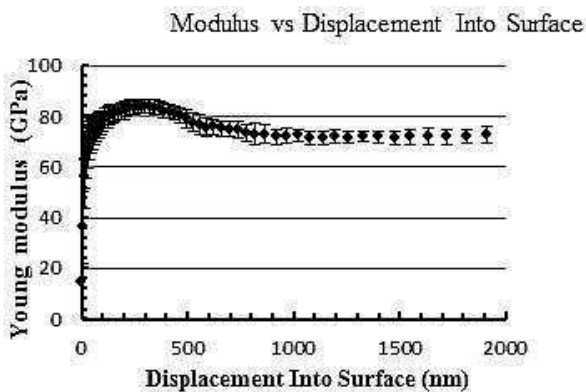


Fig. 4. Young's modulus diagram of MoS<sub>2</sub>(Ti,W) with Ti interlayer

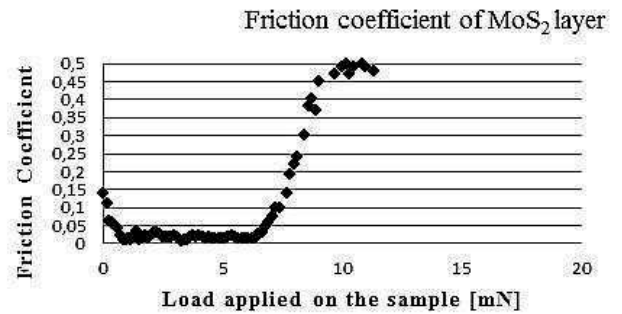


Fig. 5. The change of the average friction coefficient during the adhesion measurement test of the MoS<sub>2</sub>(Ti,W) coating

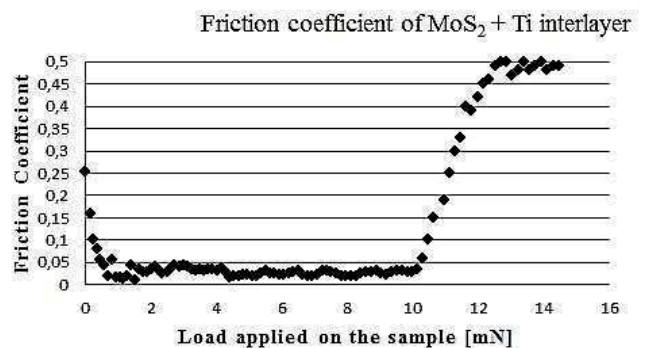


Fig. 6. The change of the average friction coefficient during the adhesion measurement test of the MoS<sub>2</sub>(Ti,W) coating with the Ti interlayer

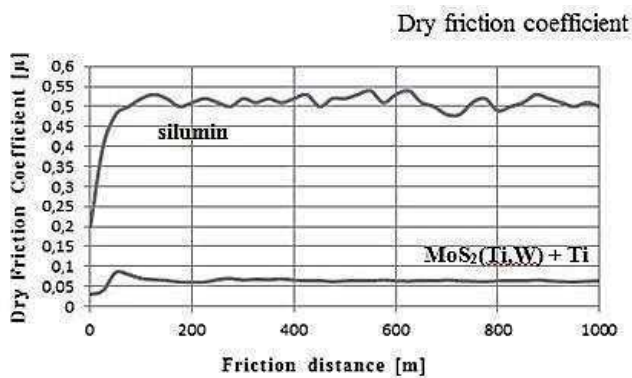


Fig. 7. Dynamic coefficients of dry friction between the bearing steel ball and (a) the Al alloy with the  $\text{MoS}_2(\text{Ti,W})$  low friction coating and the Ti interlayer, (b) silumin

Table 2.

Wear coefficients (Kw) for the silumin without coating (a) and with coating (b)

Kw wear coefficient			
(a)	silumin	(b)	silumin + $\text{MoS}_2(\text{Ti,W})$
	$2.4 \cdot 10^{-13}$		$3.2 \cdot 10^{-15}$

## 4. Conclusions

The  $\text{MoS}_2(\text{Ti,W})$  layers produced on a substrate made of modified and refined silumin significantly increase its functional properties, by way of significantly lowering the coefficient of friction (from 0.5 to 0.08) and wear (from  $2.4 \cdot 10^{-13}$  to  $3.2 \cdot 10^{-15}$ ), thus improving its mechanical properties (hardness and Young's modulus). Such good operation results would not, however, have been obtained without the Ti interlayer applied beneath the  $\text{MoS}_2(\text{Ti,W})$  coating, which decisively improves the adhesion of the produced coating to the silumin substrate. The obtained test results make it possible to apply the discussed technology for components of machines and devices made of silumins, which work under the conditions of adhesion wear.

Comparing the results of the present work with the work [16] it can be concluded that the presence of Ti, in both, main coating and as interlayer significantly increases the adhesion of  $\text{MoS}_2(\text{Ti,W})$  to the substrate than with Cr interlayer not included in the basic coating. Performance tests carried out on pistons revealed that a layer thickness of  $1.5 \mu\text{m}$  is too thin therefore a layer of  $2.5 \mu\text{m}$  thickness was manufactured.

## References

- [1] S. Yang, D.G. Teer, Investigation of sputtered carbon and carbon/chromium multi-layered coating, *Surface and Coatings Technology* 131 (2000) 412-416.
- [2] P.Eh. Hovsepian, D.B. Lewis, C. Constable, Q. Lou, Y.N. Kok, W.D. Munz, Combined steered cathodic arc/unbalanced magnetron grown C/Cr nanoscale multilayer coatings for tribological applications, *Surface and Coatings Technology* 174-175 (2003) 762-769.
- [3] Y.N. Kok, P.Eh. Hovsepian, Resistance of nanoscale multilayer C/Cr coatings against environmental attack, *Surface and Coatings Technology* 201 (2006) 3596-3605.
- [4] J. Kusiński, M. Rozmus, J.B. Bujak, Investigation of the life-time of drills covered with the anti-wear Cr(C,N) complex coatings deposited by means of Arc-PVD technique, *Journal of Achievements in Materials and Manufacturing Engineering* 33/1 (2009) 86-93.
- [5] L.A. Dobrzański, K. Gołombek, J. Mikuła, D. Pakuła, Multilayer and gradient PVD coatings on the sintered tool materials, *Journal of Achievements in Materials and Manufacturing Engineering* 33/2 (2008) 170-190.
- [6] L.A. Dobrzański, K. Lukaszewicz, K. Labisz, Structure of monolayer coatings deposited by PVD techniques, *Journal of Achievements in Materials and Manufacturing Engineering* 18/1-2 (2008) 423-426.
- [7] W. Kaczorowski, D. Batory, Carbon and titanium based layers for wood based materials, *Journal of Achievements in Materials and Manufacturing Engineering* 27/2 (2008) 381-386.
- [8] M. Clapa, D. Batory, Improving adhesion and wear resistance of carbon coatings using Ti:C gradient layers, *Journal of Achievements in Materials and Manufacturing Engineering* 20/1-2 (2007) 415-418.
- [9] L.A. Dobrzański, K. Gołombek, J. Mikuła, D. Pakuła, Cutting ability improvement of coated tool materials, *Journal of Achievements in Materials and Manufacturing Engineering* 17/1-2 (2006) 41-44.
- [10] S. Mitura, K. Mitura, P. Niedzielski, P. Louda, V. Danilenko, Nanocrystalline diamond, its synthesis, properties and applications, *Journal of Achievements in Materials and Manufacturing Engineering* 16/1-2 (2006) 9-16.
- [11] L.A. Dobrzański, L. Wosińska, K. Gołombek, J. Mikuła, Structure of multicomponent and gradient PVD coatings deposited on sintered tool materials, *Journal of Achievements in Materials and Manufacturing Engineering* 20/1-2 (2007) 99-102.

- [12] K. Gołombek, J. Mikuła, D. Pakuła, L. Żukowska, L.A. Dobrzański, Sintered tool materials with multi-component PVD gradient coatings, *Journal of Achievements in Materials and Manufacturing Engineering* 31/1 (2008) 15-22.
- [13] J. Kopač, M. Soković, Cutting properties of the PVD and CVD coatings on the ceramic substrates, *Journal of Achievements in Materials and Manufacturing Engineering* 18 (2006) 278-285.
- [14] L.A. Dobrzański, J. Mikuła, Cutting properties of the ceramic tool materials based on  $\text{Si}_3\text{N}_4$  and  $\text{Al}_2\text{O}_3$ , Proceedings of the 12<sup>th</sup> Scientific International Conference „Achievements in Mechanical and Materials Engineering” AMME’2003, Gliwice-Zakopane, 2003, 221-225.
- [15] J. Gawroński, M. Makówka, W. Pawlak, Ł. Kaczmarek, The structure and mechanical properties of nanocomposite coatings nc-WC/a-C:H, *Materials Engineering* 5 (2013) 149-152.
- [16] J. Gawroński, Ł. Kaczmarek, W. Pawlak, P. Nolbrzak, The structure and mechanical properties of nanocomposite coatings  $\text{MoS}_2$  (Ti, W) with interlayers Cr, *Materials Engineering* 6 (2014) 1-4.
- [17] M. Makówka, W. Pawlak, B. Wendler, J. Sielski, M. Kozanecki, High-temperature low-friction coating on the basis of  $\text{MoO}_3$  and Ag deposited by magnetron sputtering, *Materials Engineering* 4 (2011) 553-557.
- [18] M.B. Peterson, S.Z. Lis, S.F. Murray, Wear resisting oxide films for 900°C. Final report, anl/otm/cr-5, US Department of Energy, 1994.
- [19] S.M. Aouadi, B. Luster, P. Kohli, C. Muratore, A. Voevodin, Progress in the development of adaptive nitride-based coatings for high temperature tribological applications, *Surface and Coatings Technology* 204 (2009) 962-968.
- [20] M. Makówka, T. Moskalewicz, K. Włodarczyk, B. Wendler, Low-friction and wear-resistant nanocomposite coating type nc-Ci/a-C and nc-Ci/a-C:H, *Materials Engineering* 4 (2010) 1091-1095.
- [21] V. Singh, J.C. Jiang, E.I. Meletis, Cr-diamondlike carbon nanocomposite films: Synthesis, characterization and properties, *Thin Solid Films* 489 (2005) 150-158.
- [22] G. Gassner, J. Patscheider, P.H. Mayrhofer, S. Sturm, C. Scheu, C. Mitterer, Tribological properties on nanocomposite  $\text{CrCx/a-C:H}$ , *Thin Films Tribology Letters* 27 (2007) 97-104.
- [23] Z.Q. Qi, E.I. Meletis, Mechanical and tribological behaviour of nanocomposite multilayered Cr/a-C thin films, *Thin Solid Film* 479 (2005) 174-181.
- [24] K. Włodarczyk, M. Makówka, P. Nolbrzak, B. Wendler, Low friction and wear resistant nanocomposite nc-MeC/a-C and nc-MeC/a-C:H coatings, *Journal of Achievements in Materials and Manufacturing Engineering* 37 (2009) 354-360.
- [25] W. Pawlak, R. Atraszkiewicz, P. Nolbrzak, B. Wendler, Low friction coating  $\text{MoS}_2$  (Ti, W) deposited by magnetron sputtering on the nitrated and HSS Vanadis 23, *Materials Engineering* 4 (2010) 1157-1161.